EDGEWOOD

CHEMICAL BIOLOGICAL CENTER

U.S. ARMY SOLDIER AND BIOLOGICAL CHEMICAL COMMAND

ECBC-TR-160

ROBOTIC SYSTEM DESIGN FOR PREPARATION OF MILITARY CHEMICAL AGENT STANDARDS AND EXTRACTION OF MILITARY CHEMICAL AGENTS IN ENVIRONMENTAL SAMPLES

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13. ABSTRACT *(Maximum 200 words)*

This report describes a robotic system design for preparing military chemical agent standards' extraction of military chemical agents in environmental samples. Several factors have led to the application of robotics for handling and preparing very toxic chemical agents. First, these samples range from high-to-low volatility and use highly toxic chemicals requiring special handling. Careful and well-planned work with attention to caution, cleanliness, and the use of all possible protective devices is required. Secondly, a large number of samples are processed, making it desirable to use an automated system. Thirdly, repeatability, precision, and accuracy are needed for an acceptable quality assurance program. To accomplish the automation, a CRS *PLUS*-464 Robotic articulated arm (Hudson Control Group, Springfield, NJ) with various stations enclosed within a fumed hood was designed for ECBC chemical surety operations. Use of this integrative technique reduces the time frame for preparing and extracting samples for the preparation of sample/standards containing military chemical agents. Some additional benefits of the robotic system application include reliable documentation, higher productivity, safer laboratory conditions, and future expandability.

14. SUBJECT TERMS
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PREFACE

The work described in this report was authorized under Project No. 622622, CASARM Program. The work was started July 1997 and completed November 1999.

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ROBOTIC SYSTEM DESIGN
FOR PREPARATION OF MILITARY CHEMICAL AGENT STANDARDS AND EXTRACTION OF MILITARY CHEMICAL AGENTS IN ENVIRONMENTAL SAMPLES

1. INTRODUCTION

1.1 Objective.

The objective of this report is to describe a robotic system designed and developed for preparing military chemical agent (MCA) standards, extracting, and analysis preparation of MCA's in environmental samples. This system assists the worker in providing reliable documentation, repeatability in every day routine analysis, higher productivity, a safer work environment, and for future expandability in the laboratory.

1.2 Background.

The Chemical Analysis Chemistry Team (CAT), Research and Technology Directorate, U.S. Army Edgewood Chemical Biological Command (ECBC), mission includes preparation of chemical agent standard analytical reference materials (CASARM), and extraction and analyses of environmental samples suspected of being contaminated with chemical agents. These standards and samples are very toxic and require trained professionals with specialized training when conducting experiments. Because of the compounds' toxicity and the routine experimental approach, a system is needed to increase worker safety while providing repeatability during sampling and analysis.

There are several military installations that have identified a large quantity of chemical waste materials that need to be disposed of in a safe and environmentally acceptable manner. Since that waste is contaminated with chemical agent and related products, qualitative identification and quantitative analysis of any potential MCA contamination are essential for safe disposal operations. Prior to release from government control ultimately for proper disposal, environmental samples must be analyzed to be sure they do not contain levels of MCA above the quantity considered safe for the soldiers consumption. According to the TB-MED 577, dated March 1986, these drinking water standards for soldiers' consumption levels for MCAs are as follows: 200 parts per billion for Bis (2-chloroethyl) sulfide, 20 parts per billion for Isopropyl methylphosphonofluoridate (sarin), and pinacolyl methylphosphonofluridate (soman).[1] To accomplish the task of quantitation, standard solutions are needed for calibrations of instruments and various standard addition techniques prior to quantitative analysis. Extraction of environmental samples is the primary approach for solid/liquid matter to determine MCA and related compound composition. This standard approach to sample preparation, extraction, and analysis takes 60 minutes. If the sample preparation and extraction process is not completed within the predetermined holding times, the samples decompose and the sample preparation process must start over again on a newly collected sample. This increases the overall program/ project cost while preventing the laboratory from meeting critical reporting schedules.

[1] U.S. Army Technical Bulletin MED 577, March 1986.

The application and design of the state-of-the art automated laboratory robotic system is intended to eliminate the ever-increasing lag time between sample preparation, sample extraction, and sample analysis time. The robotic system's capabilities offer a flexible approach to the preparation and extraction of liquid/solid samples. The system performs the same manipulation of a human, making sample preparation possible with a better precision and accuracy. The robotic approach will cut cost, because tasks will not have to be repeated due to the system's mechanical precision, and this approach will provide workers with a safer working environment. This is accomplished because the system automatically weights out the sample, performs the dilution, mixes the sample, performs liquid/solid extractions, and transfers an aliquot of the sample to a gas chromatography (GC) vial. Since the mechanical arm can be programmed for various tasks, the repeatability, precision, and accuracy is more consistent than a human. The robotics application was chosen because current experimental procedures are labor-intensive in which samples are highly toxic, requiring special handling, and its eventual application would represent a means of reducing the workload for the laboratory worker. Additional benefits of the application of the robotic system include reliable documentation, higher productivity, safer laboratories, and future expandability.

2. EQUIPMENT

2.1 Software.

The CRS *PLUS*-464 Robotic System has a standard 16-bit microprocessor based master controller, resident robotic automation programming languages (RAPL II), six DC servo amplifiers, arm power supply, voltage regulators, and five intelligent servo axis cords. RAPL II is an automated oriented line structured language designed to facilitate the design of applications of the robotic system. The system computer communicates to the robot controller each task to perform and receives a completion message in return. The system has a teach pendant that manually controls the robot system arm, limb, and align functions, gripper functions, and three additional degrees of freedom. RAPL II uses English-like commands to provide a user-friendly interface for the operator. It has an MS-DOS operating system with GW-BASIC programming language.

2.2 Hardware.

A CRS *PLUS*-464 Robotic system was design by Hudson Control Group (Springfield, NJ) under contract No. DAA05-92-B-0032 for ECBC. The key features of this robotic system are CRS *PLUS*-464 Robotic arm (Figure 1), ampoule holding station (Figure 2), ampoule breaking station (Figure 3), capping station (Figure 4), agent funnel rack (Figure 5), agent pipetting station, and environmentally controlled fume hood (Appendix A). These key features of the robotic system allow all sample handling techniques to occur within a controlled environment. Therefore, toxic and hazardous operations can be done without human exposure to any hazardous or toxic materials. The CRS *PLUS*-464 articulated robotic arm moves and simulates movements of a human inside its enclosed fumed hood. The arm manipulates ampoules of MCA from rack to ampoule breaking station, test tubes, and bottles from the rack to both an analytical

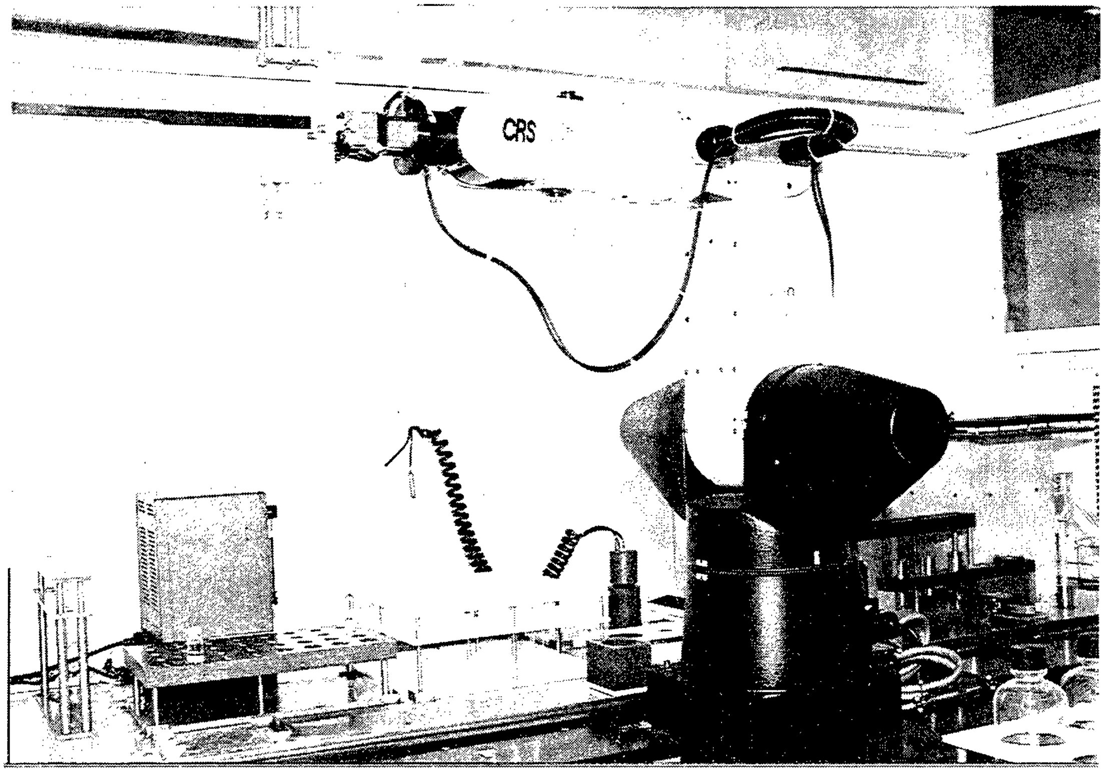


Figure 1. CRS *PLUS*-464 Robotic Arm

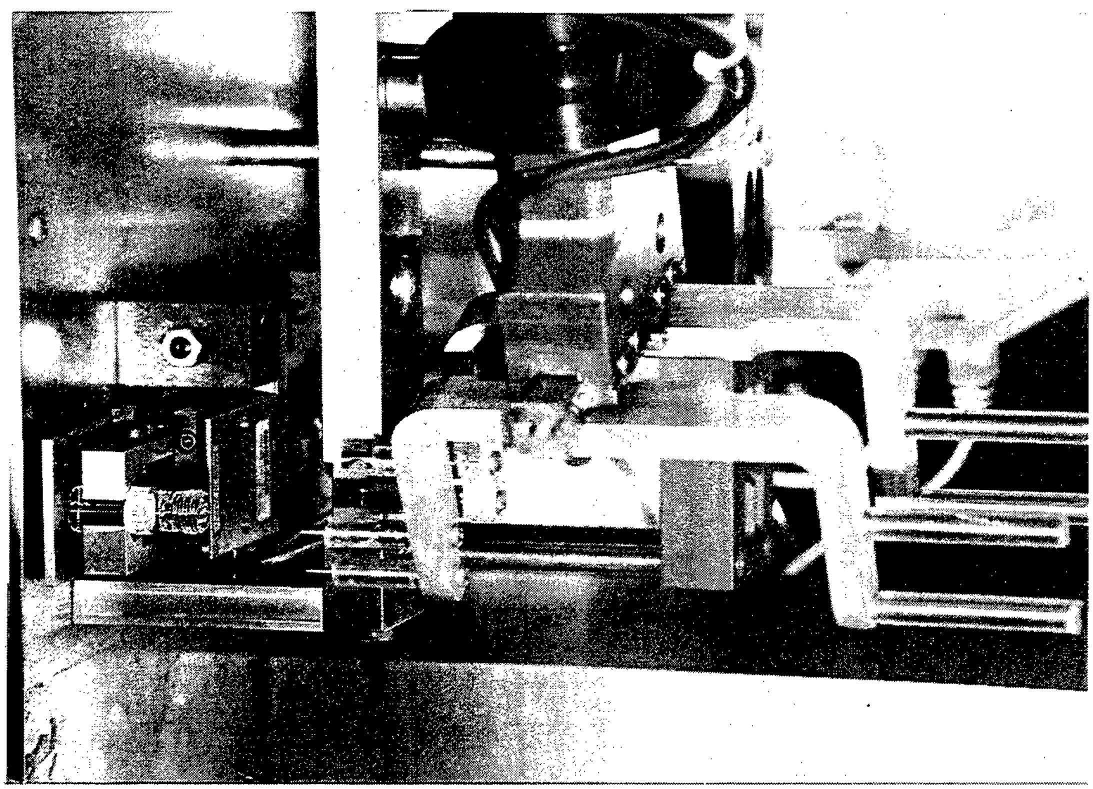


Figure 2. Ampoule Holding Station

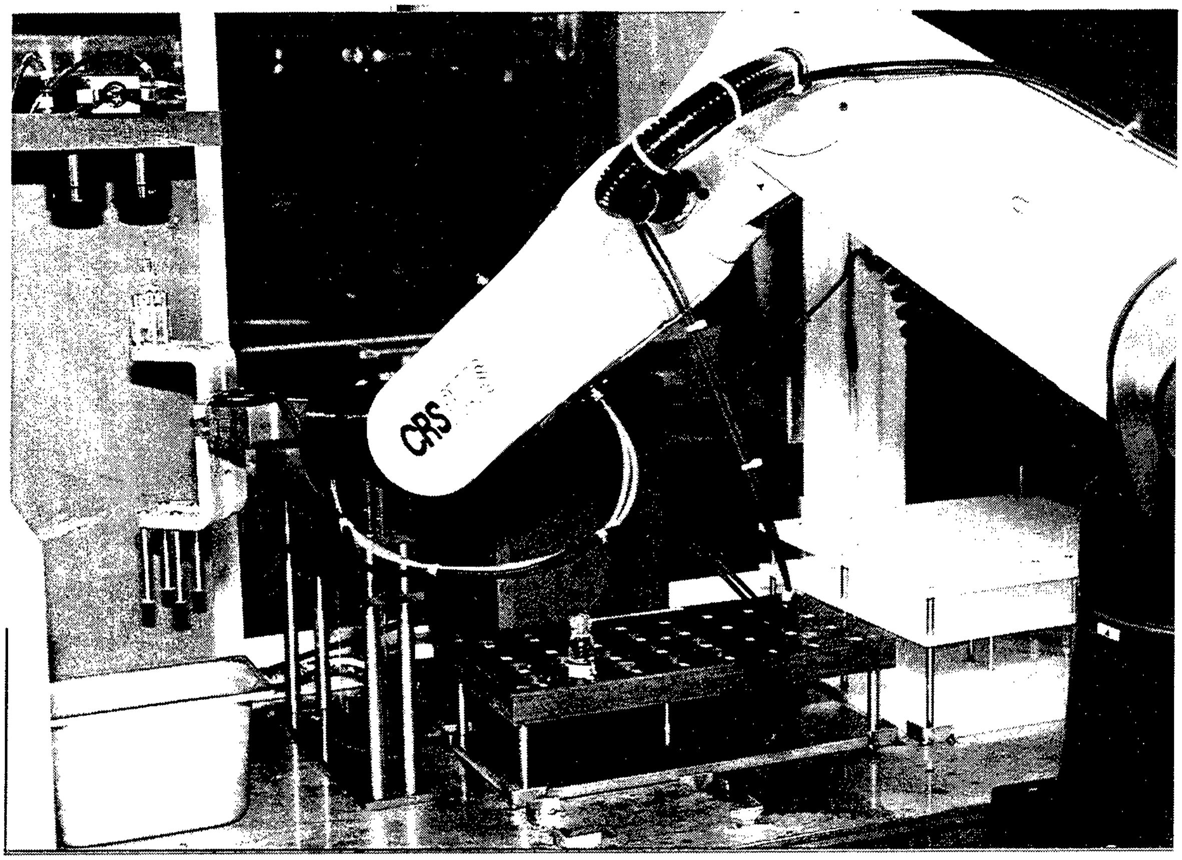


Figure 3. Ampoule Breaking Station

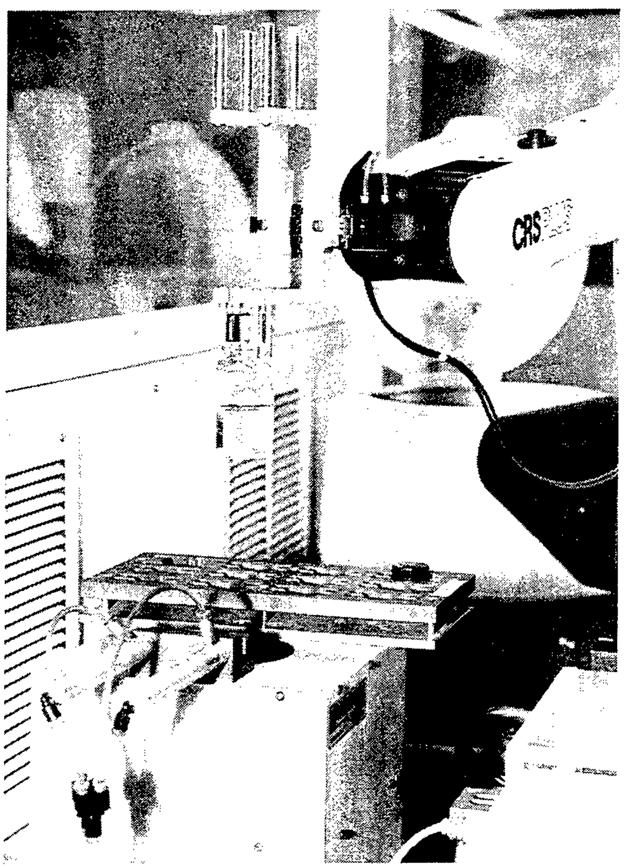


Figure 4. Capping Station

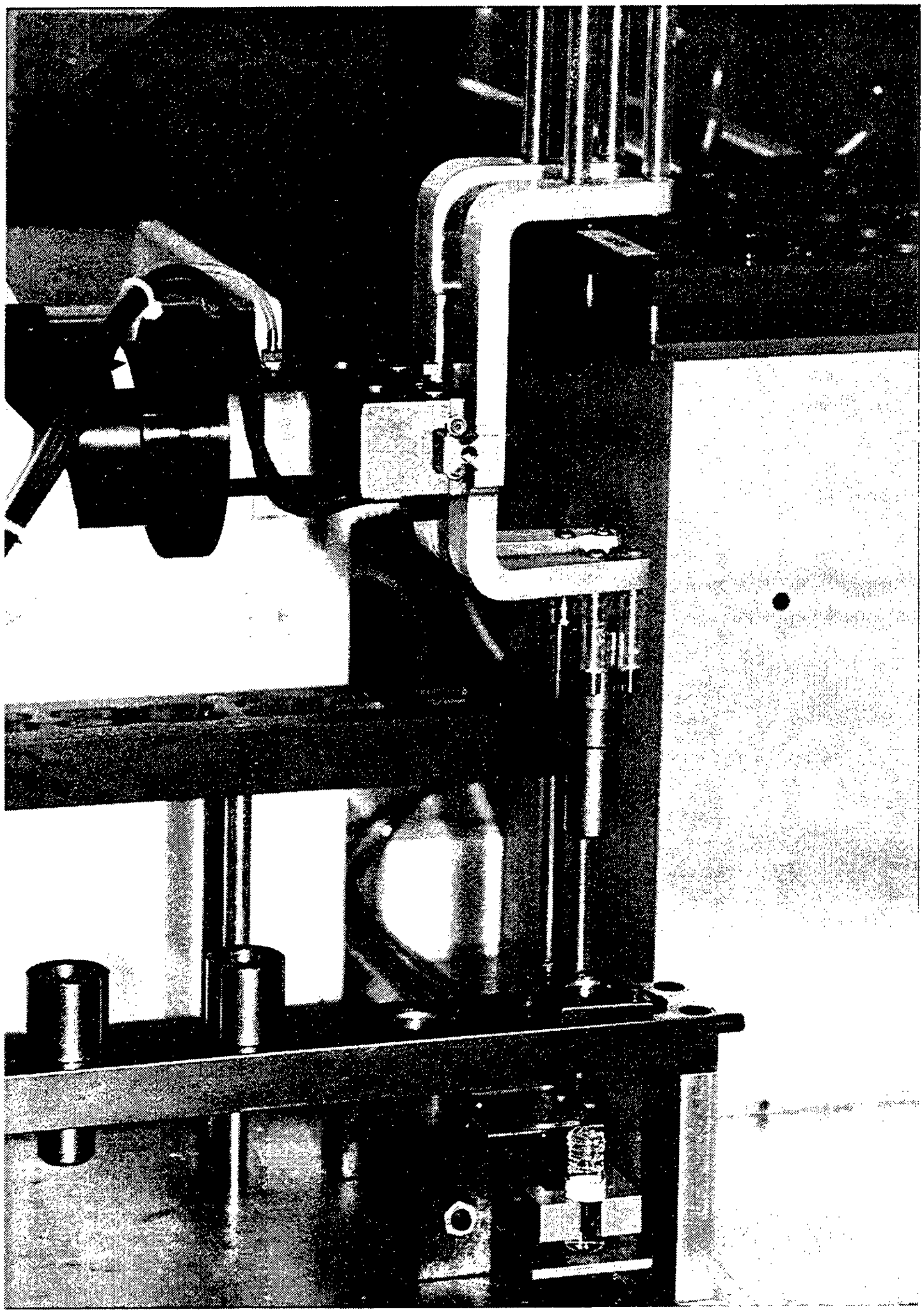


Figure 5. Agent Funnel Rack

balance and a vortex station. A series of syringes is employed to add appropriate solvent gravimetrically to bottles in the balance station.

3. PROCEDURE

3.1 Preparation of Standards.

Analytical procedures routinely include sample weighing, sample dilution, standard preparation, solvent extraction of the analyses from the matrix, liquid: liquid extraction, solid phase extraction, and concentration analyses. These factors are ideally suitable for robotic capabilities. The robotic equipment selected will perform this operation needed for flexibility to perform a variety of movements and operations, handle various labware, and provide precise reliable data. The standard solutions of MCA's are prepared from neat CASARMs, which must meet a purity criterion of 95% or better.[2] The complete robotic system is shown in Appendix B. In preparation of a standard solution of MCA, the analyst will place the neat MCA ampoule into the ampoule holding secondary dilution bottles into the bottle output rack. The analyst will close the fume hood door and activate the control button for the robot to start the standard preparation process. The robotic arm (Figure 1) retrieves an MCA ampoule from the ampoule holding rack (Figure 2) and places it in the ampoule breaking station (Figure 3). The robotic arm retrieves the broken ampoule from the ampoule breaking station (Figures 6 and 7) and places the ampoule into the ampoule pipette's holding station (Figures 2 and 8). The robot will retrieve an ampoule pipette funnel (Figure 5) and place it into the funnel holder to pipette MCA out of the ampoule. The arm will carry the bottle to the solvent dispense station and fill the bottle with ½ of the desired amount of solvent. The arm will retrieve the bottle from balances, place a cap on the bottle, and then mix the solution. The arm will place the bottle onto the balance and dispense the desired amount of agent from the MCA ampoule into primary bottle. The amount of agent dispensed will be recorded and saved to computer. The arm will dispense pipette into waste container, retrieve primary bottle, fill with desired amount of solvent to mark, place on capping station, cap mix, and place the container in the rack. The same procedure will be done for the secondary bottle. The final concentration of the primary standard solution will be 3% higher than the secondary standard solution. A serial dilution process will be done using the primary (stock A) solution standard. The robot will get the primary solution and do serial dilution standards of 8 ml/10 ml, 5 ml/10 ml, 4 ml/10 ml, 3 ml/10 ml, and 2 ml/10 ml. This is a CASARM dilute standard curve. The bottles will be capped. The solution will be mixed well, and the robotic arm will transfer an aliquot of the solutions into GC vials for analysis. Once the solutions are prepared, analyzing the prepared solution on the GC generates a calibration curve. The data is collected and analyzed by the analyst. A typical calibration curve is generated using standards that range from 1-10 mg/ml for nerve and blister agents. For concentrations lower than 1-10 mg/ml (drinking water level, microgram/milliliter range), standards are prepared using the Drinking Water Dilution process (Appendix B).

[2] Vickers, E.L., Sumpter, K.B., *Aggravated Storage Determination of Neat CASARM in Glass Ampoules Stored at 70°for Two-weeks,* ERDEC-TR-304, U.S. Army Edgewood Research, Development and Engineering Center, Aberdeen Proving Ground, MD, January 1994.

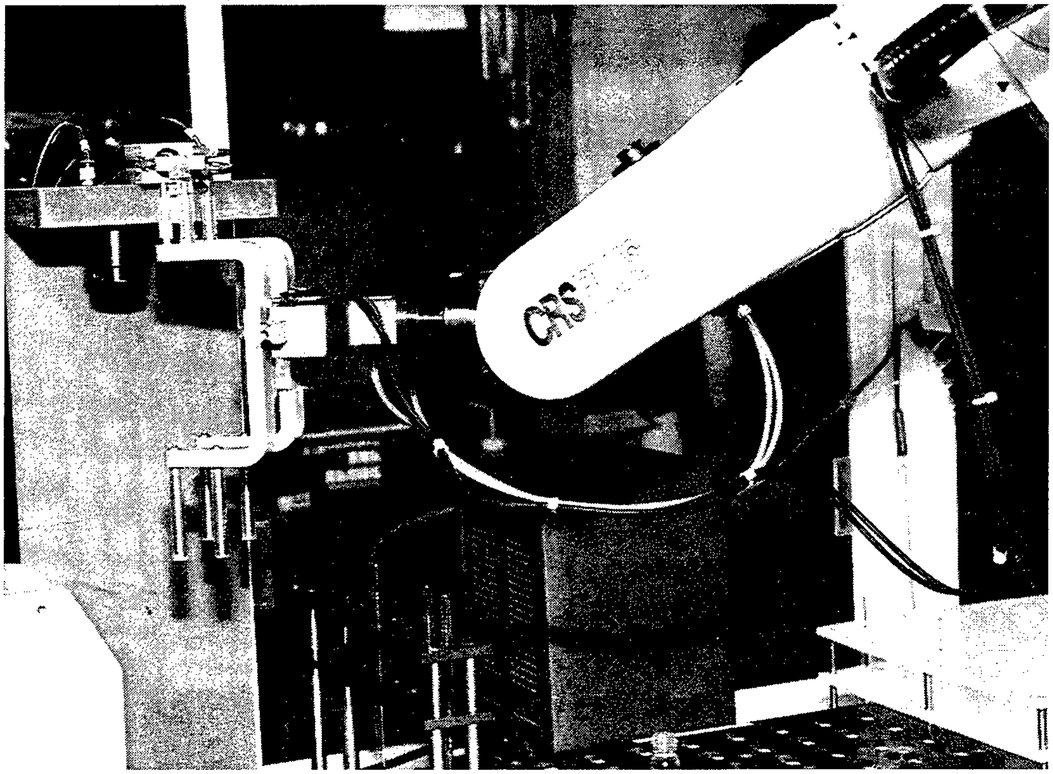


Figure 6. Robotic Arm Retrieving Broken Ampoule

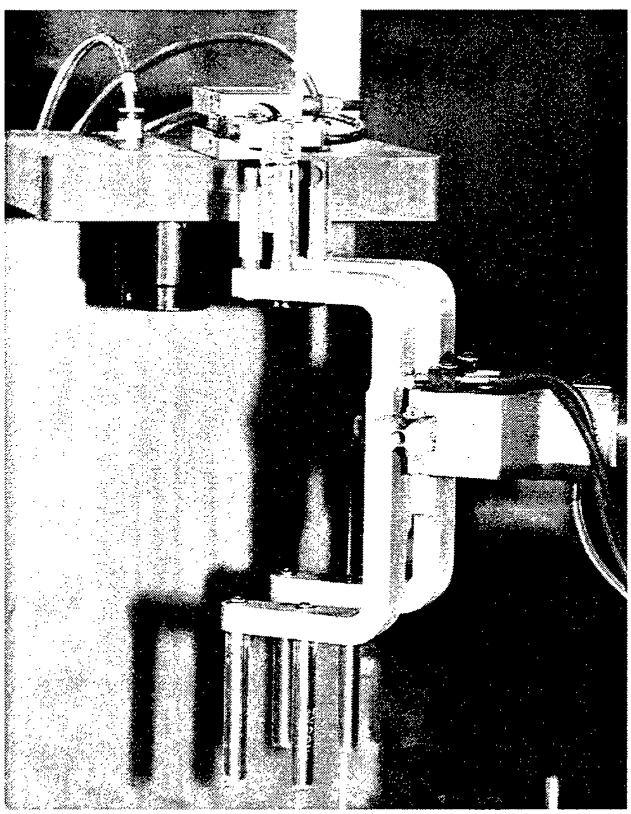


Figure 7. Robotic Arm Holding Broken Ampoule

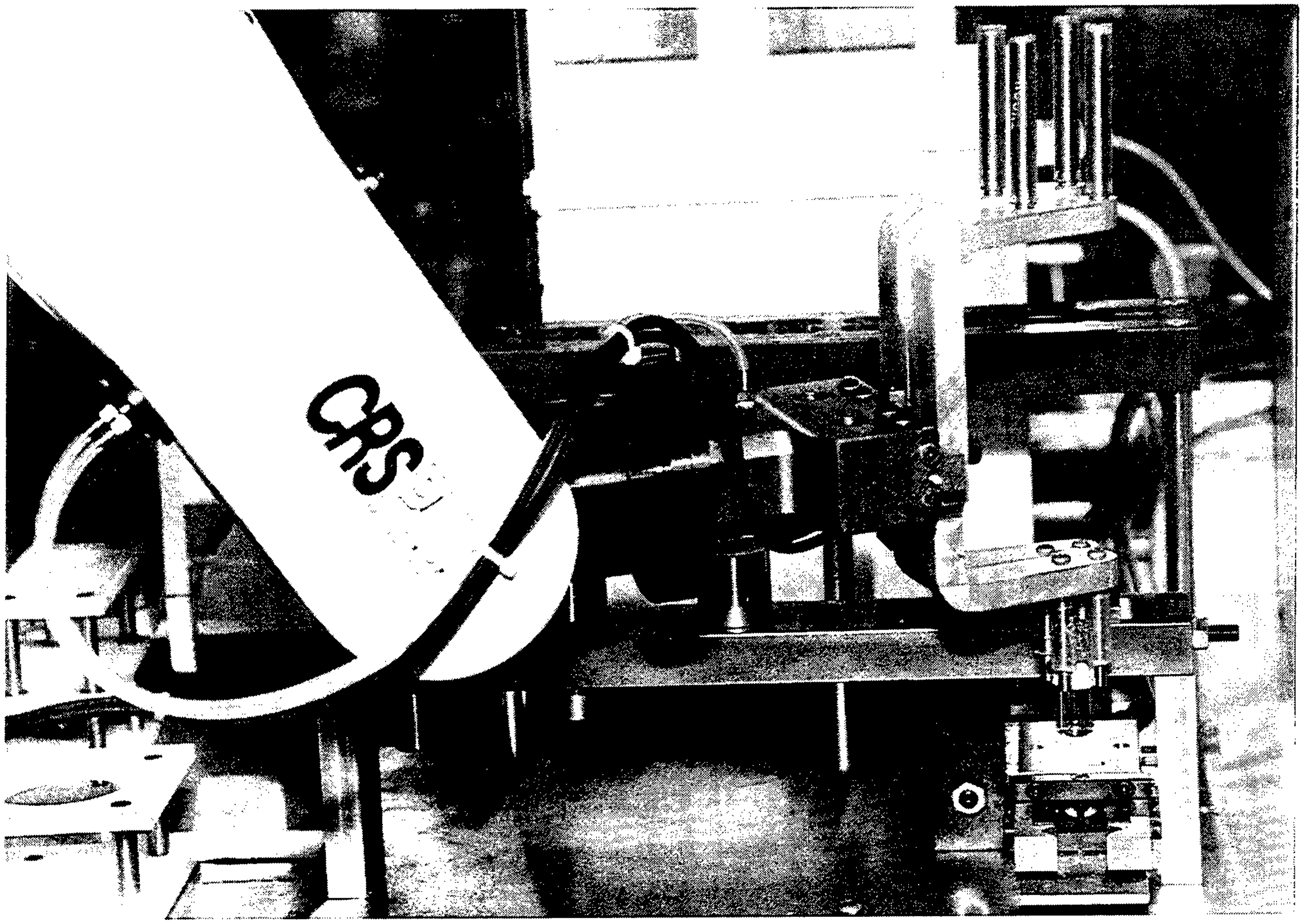


Figure 8. Ampoule Pipette's Holding Station

3.2 Extraction.

If the sample matrix is a solid, it is weighed out, mixed, vortex and split into two aliquot (spiked and unspiked) samples (Appendix E). In the case of aqueous samples, the pH is taken and then the sample is filtered. For this particular case, the extraction solution is first added, followed by the spiking solution, to the spiked samples, and finally extracted (Appendix F). The resulting extracts are analyzed on gas chromatograph flame photometric detector (GC/FPD) in the phosphorus or sulfur mode.

3.3 Robotic Configuration.

The CRS *PLUS*-464 is an articulated robot arm consisting of five components; base, shoulder, upper arm, lower arm, and wrist. The position repeatability of the arm is ± 0.002 inches along the total arm of 22 inches. The robotic system has a 16-bit microprocessor-based controller, resident RAPL programming language, six DC servo amplifiers, an arm power supply and voltage regulator, and five intelligent servo axis cards. The robotic system is mounted on a table enclosed within a fume hood 8 feet 6 inches by 5 feet 6 inches. The system consists of the following stations and equipment: ampoule-breaking station, liquid dispensing station, capping station, ampoule input rack, pipette tip holder, auto sampler vial holder, Mettler balance, stainless steel drip pan, sonicator, vortex and centrifuge (Appendix B).[3] The system was designed to be operated with the doors on the fumed hood closed and alarm sensors activated (Appendix A).

Fume Hood

The robotics enclosure is 66 inches x 102 inches. It is an environmentally controlled fume hood mounted on a table constructed of aluminum that has an epoxy coated painted finish, which is chemical resistant and easy to clean. The hood has a one piece plexiglas plenum with ¼-inch plexiglas sliding doors, and four doors on two tracks (front and rear). There are eight proximity contact sensors for each door to activate an alarm if a door opens during operation. It is equipped with both visual and audio alarm and minihelic gauge. It has an air sensoring device and a warning light system rated for 120-vac supply to provide and monitor airflow for correct face velocity. The fume hood is designed to maintain 150 ± 30-LPM face velocity. It has an 8-6 foot track for the robotic arm.

4. SUMMARY/CONCLUSIONS

This system is good for labor-intensive procedures, because it can perform these tasks over and over again, 24 hours per day/7 days a week. The robotic system is safe for the worker. It will reduce the hazardous chemical contact times for worker exposure by 90%. The only time the worker has to handle the toxic materials is during initial transfers of the materials from their storage area to the fume hood, and when these chemicals are packaged up and returned to the storage area. In the past, the worker had to transfer the agent from the storage room, open the toxic material, perform dilutions, extractions, transfer material into GC vials, and return the

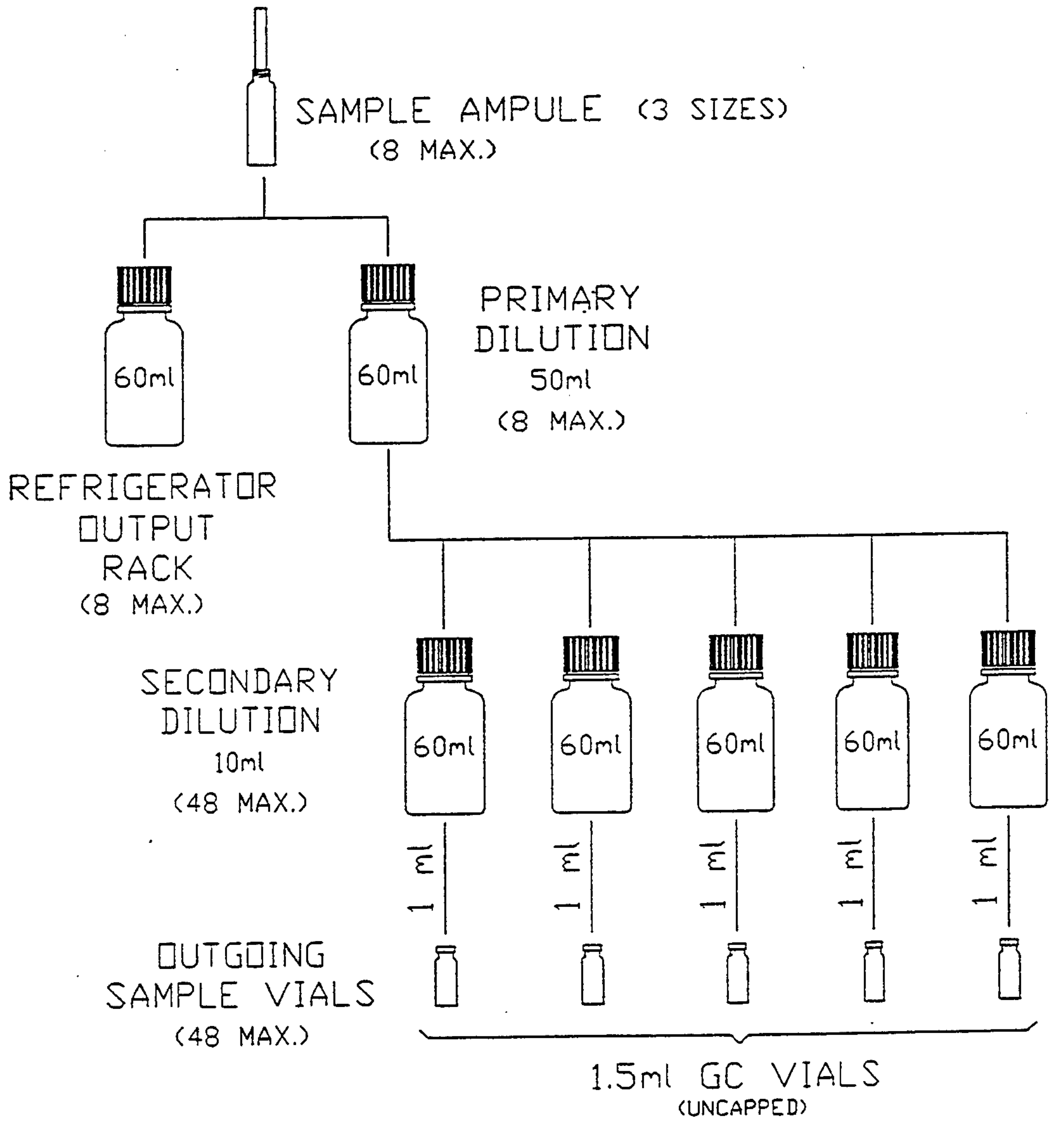
[3] Hudson Control Group, "U.S. Army ERDEC Robotic System Diagram," (Contract No. DAAA05-92-B-0032), 1994.

packaged material to the storage area. With this system, the tasks of performing dilutions, extracting, transferring chemical material to GC vials and handling toxic material by hand with protective gear for more than 45 minutes is eliminated. The elimination of that task allows the worker to be more productive in other areas of the experiment and work in a safer environment.

Implementation of the state-of-the-art robotic system for the analysis of military chemical agents in environmental samples will eliminate the ever-increasing lag time between sample preparation, sample extraction, and GC analysis. The use of this integrative technique will reduce the labor-intensive procedure faced by workers. The system is unique for performing the routine tasks performed over and over. This system will cut workers' contact time to very toxic materials by 90% and increase their productivity in other areas of work. Workers will not be tired from the repetitive, labor-intensive procedures of routine sample preparation. Additional benefits of the application of the robotic system include reliable documentation, safer laboratories, and future expandability. Pre-operational and start-up test are planned for this system in the near future. Afterwards, experiments shall be performed and data gathered to illustrate overall improvements.

APPENDIX A

CASARM DILUTION



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TITLE:

ROBOT SYSTEM PROCESS DIAGRAM SH.1

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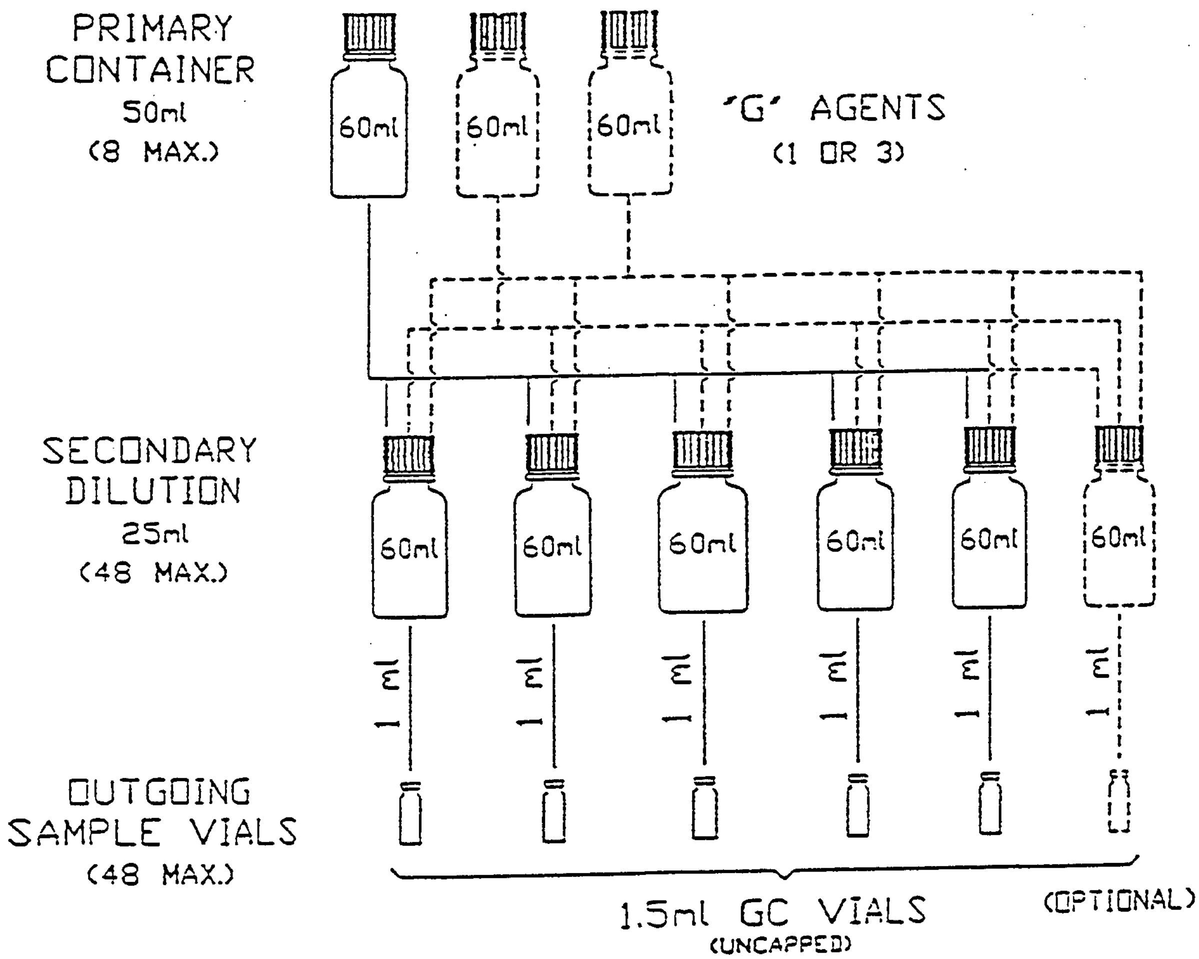
APPENDIX B

DRINKING WATER DILUTION



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ROBOT SYSTEM PROCESS DIAGRAM SH.2

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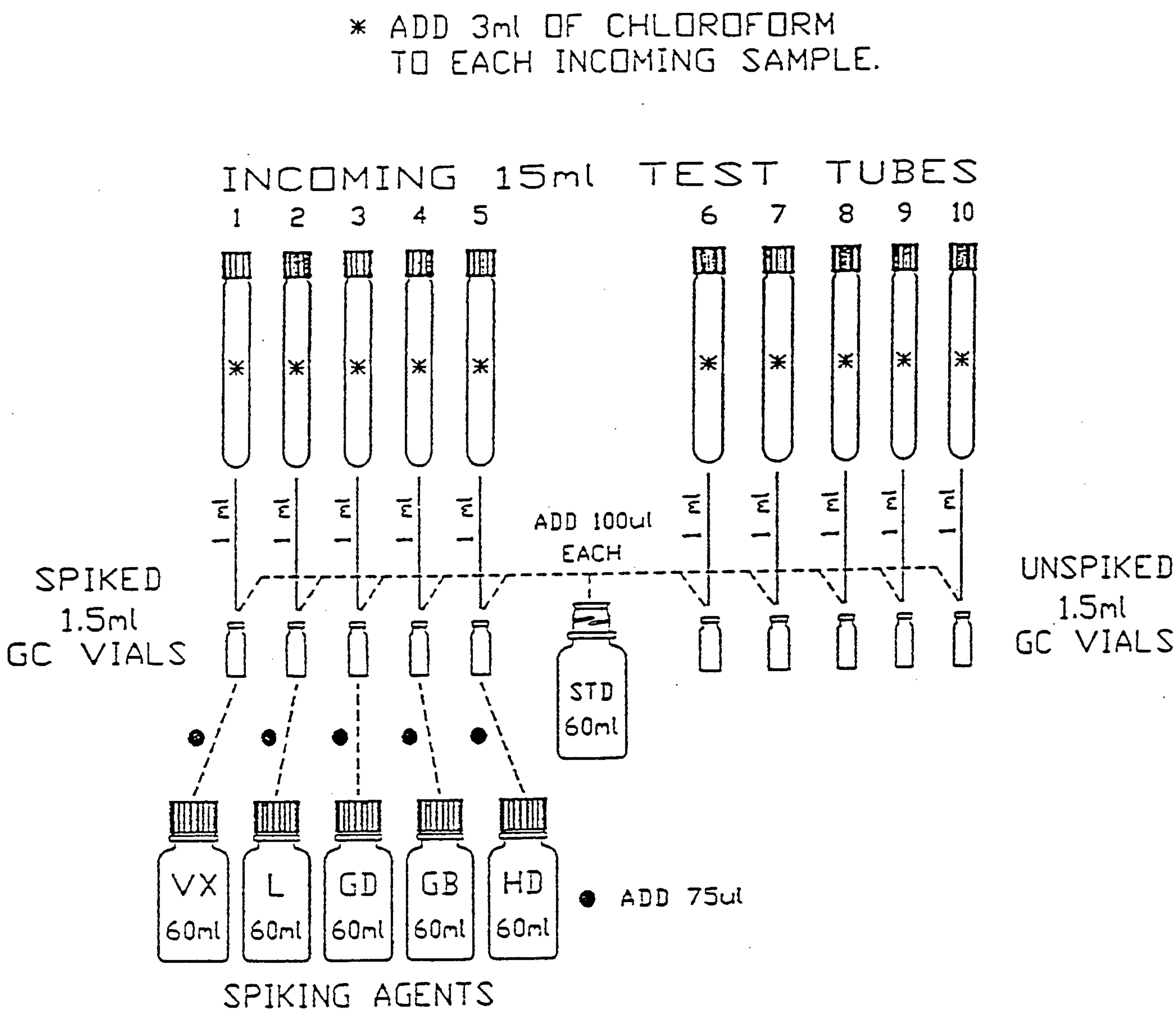
APPENDIX C

EXTRACTION - SOLIDS



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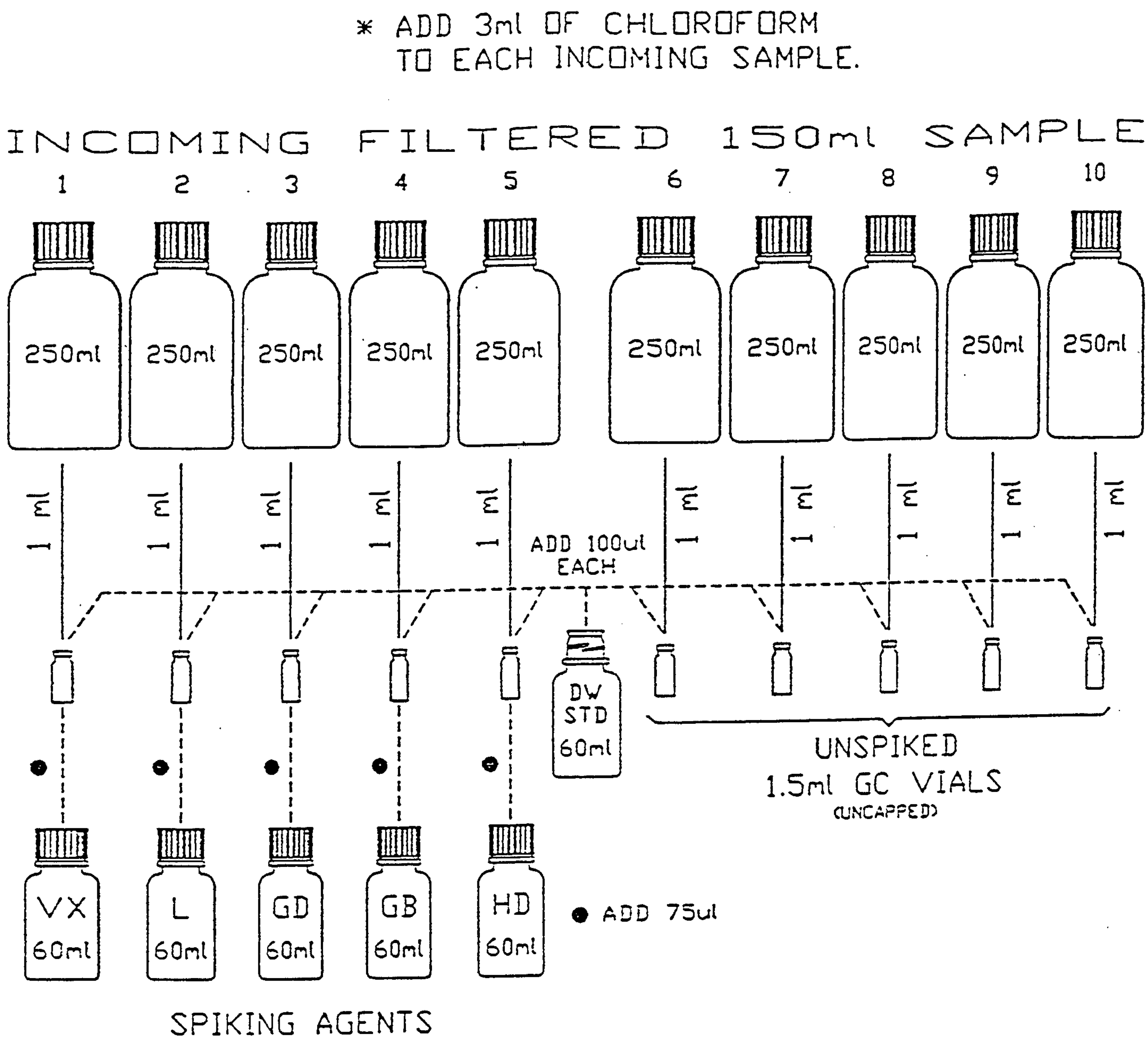
APPENDIX D

EXTRACTION – LIQUIDS



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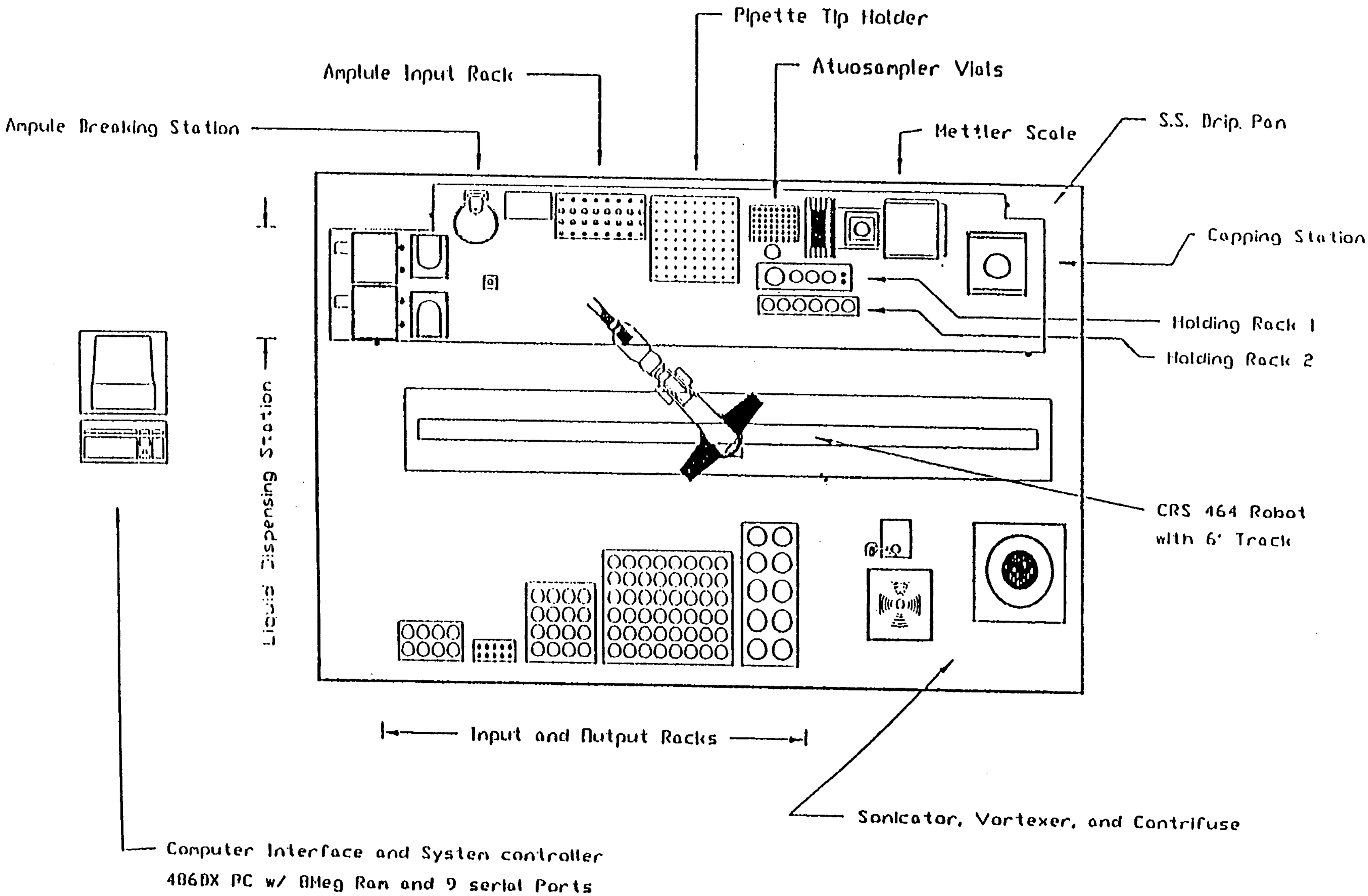
APPENDIX E

ROBOT TABLE LAYOUT



Note: Entire Table is under Fumehood (not shown)

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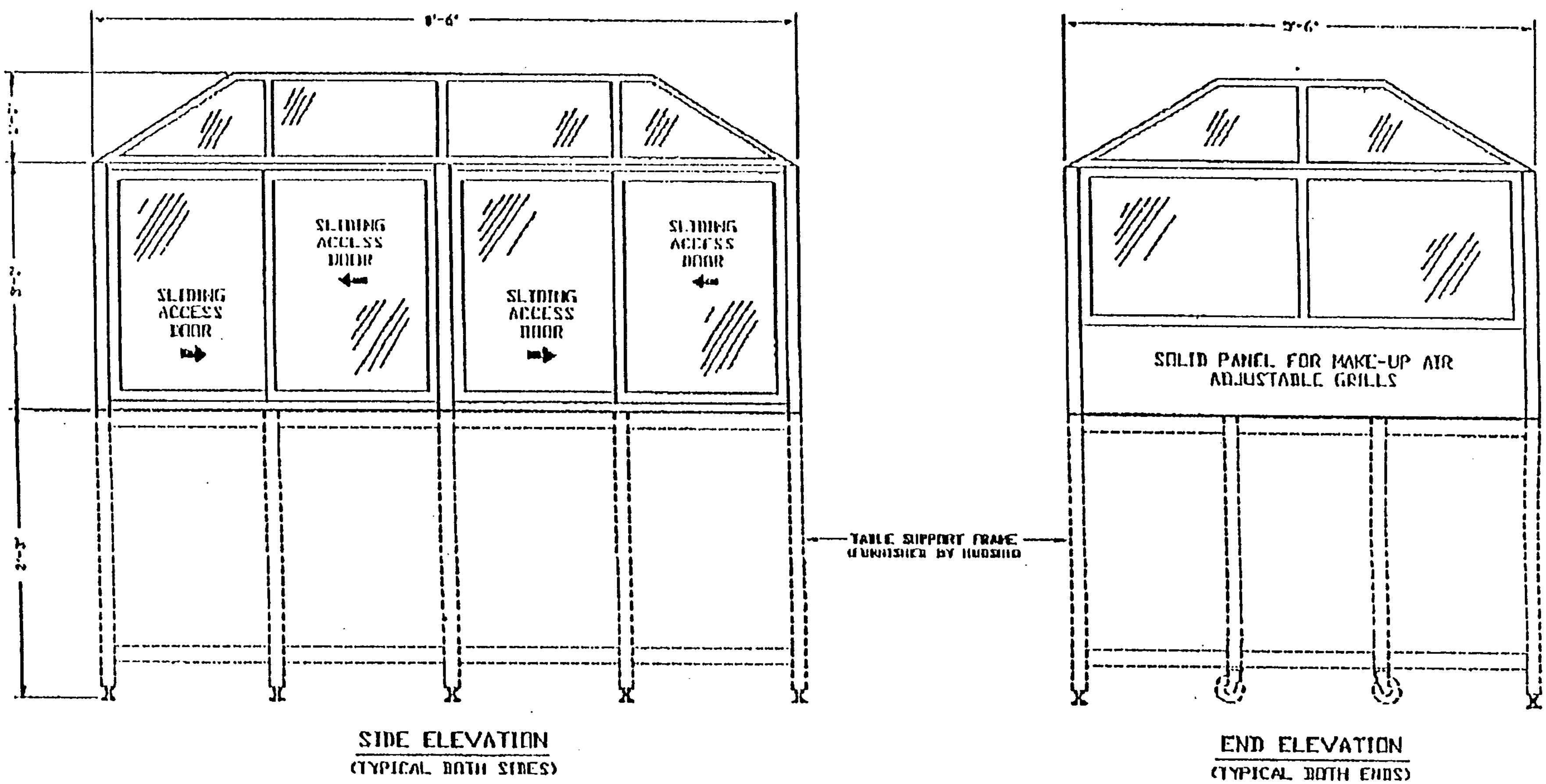
APPENDIX F

ROBOT TABLE FUME HOOD CONCEPTUAL LAYOUT



NOTES:

1. FUME HOOD MAIN SUPPORT STRUCTURE, DOOR FRAMES, FIXED PANELS AND GABLED ROOF ARE TO BE CONSTRUCTED OF ALUMINUM WITH AN EPOXY COATED POWDERED PAINT FINISH.
2. END PANELS ARE TO HAVE A SOLID BOTTOM AREA TO ACCOMMODATE ADJUSTABLE GRILLS TO CONTROL MAKE-UP AIR FLOW.
3. FUME HOOD INTERNAL ATMOSPHERE CONTAINS A FORMIC ACID VAPOR.
4. THE OUTGOING AIR WILL BE FILTERED THROUGH TWO(2) HEPA FILTERS (1 PARTICLE, 1 CHARCOAL) (FURNISHED BY OTHERS)
5. AN AIR SENSING DEVICE AND WARNING LIGHT SYSTEM, RATED FOR 120VAC SUPPLY, ARE TO PROVIDED TO MONITOR AIR FLOW FOR CORRECT FACE VELOCITY.
6. FUME HOOD WILL BE DESIGNED TO MAINTAIN A 150 +/- 30 FPM FACE VELOCITY.
7. ACCESS DOORS WILL BE CLOSED DURING NORMAL OPERATION. OCCASIONAL OPENINGS MAY OCCUR TO REPLACE SYSTEM COMPONENTS.
8. DOOR SENSORS, RATED FOR 120VAC SUPPLY, WILL BE PROVIDED TO ASCERTAIN THAT EACH DOOR IS CLOSED.

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TITLE:
ROBOT TABLE FUME HOOD
CONCEPTUAL LAYOUT

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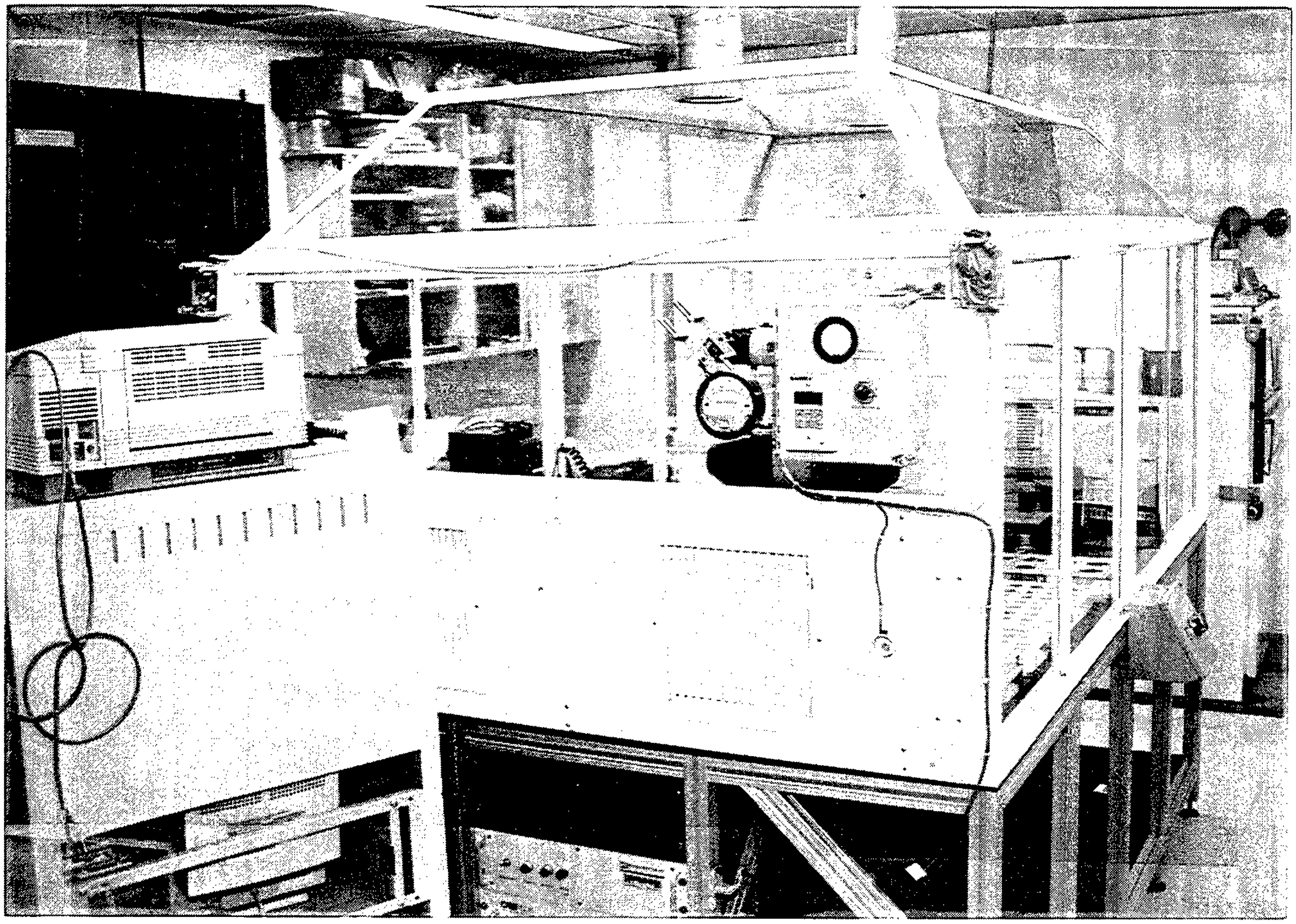

Robot Table Fume Hood Layout